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Studies of Tempering Behavior of Steel X-9165 of N-A-X 9100 Series

James A. Ndulue

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STUDIES OF TEMPERING BEHAVIOR OF STEEL

X-9165 OF N-A-X 9100 SERIES

by

James A. Ndulue

A Thesis

Submitted to the Department of Metallurgy
in Partial Fulfillment of the
Requirements for the Degree of
Bachelor of Science in Metallurgical Engineering

MONTANA SCHOOL OF MINES

BUTTE, MONTANA

May 15, 1953

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PART I: INTRODUCTION

Purpose of Investigation

The purpose of this investigation is to study the behavior of steel X-9165, when subjected to different tempering treatments, and to establish the time-temperature relationships during each tempering treatment. Through hardness measurements, it will also be possible to determine if the theoretical postulates of tempering apply to this steel. The steel used for this investigation was supplied by the Great Lakes Steel Corporation of Detroit, Michigan, to previous students at Montana School of Mines who worked out its Time-Temperature-Transformation Curves. The chemical composition of the steel, as given by the manufacturer, is as follows:

Carbon	0.58	per cent	
Manganese	0.79	"	"
Silicon	0.80	"	"
Chromium	0.74	"	"
Molybdenum	0.22	"	"
Zirconium	0.14	"	"

Brief History of Steel-Treating Process

One of the reasons why steel is the most versatile of all the metals available for engineering applications is because of the wide range of properties that can be obtained from it by proper heat treatment. Generally, two basic operations are necessary to bring out the required properties -- heating and cooling. The ancients found that steel (then called iron) which was soft and ductile, could be made extremely hard by heating to a high temperature and quenching in a liquid medium. They also knew that steel was easier to shape and form in the heated condition. Charcoal was used for heating and quenching was obviously done in water. Unintentionally, they "discovered" the process of improving the properties of iron and thus paved the way for the art of blacksmithing and metal working.

A great deal of superstition grew out of the blacksmiths' inability to explain the high strength of a quenched steel structure, and the high ductility of a heated one. The improved properties of various steels were thus attributed to some magical substance in the quenching medium and the steel. Urine and the blood of a fat slave were thus the quenching media that could produce such remarkable properties in the Damascus Sword!

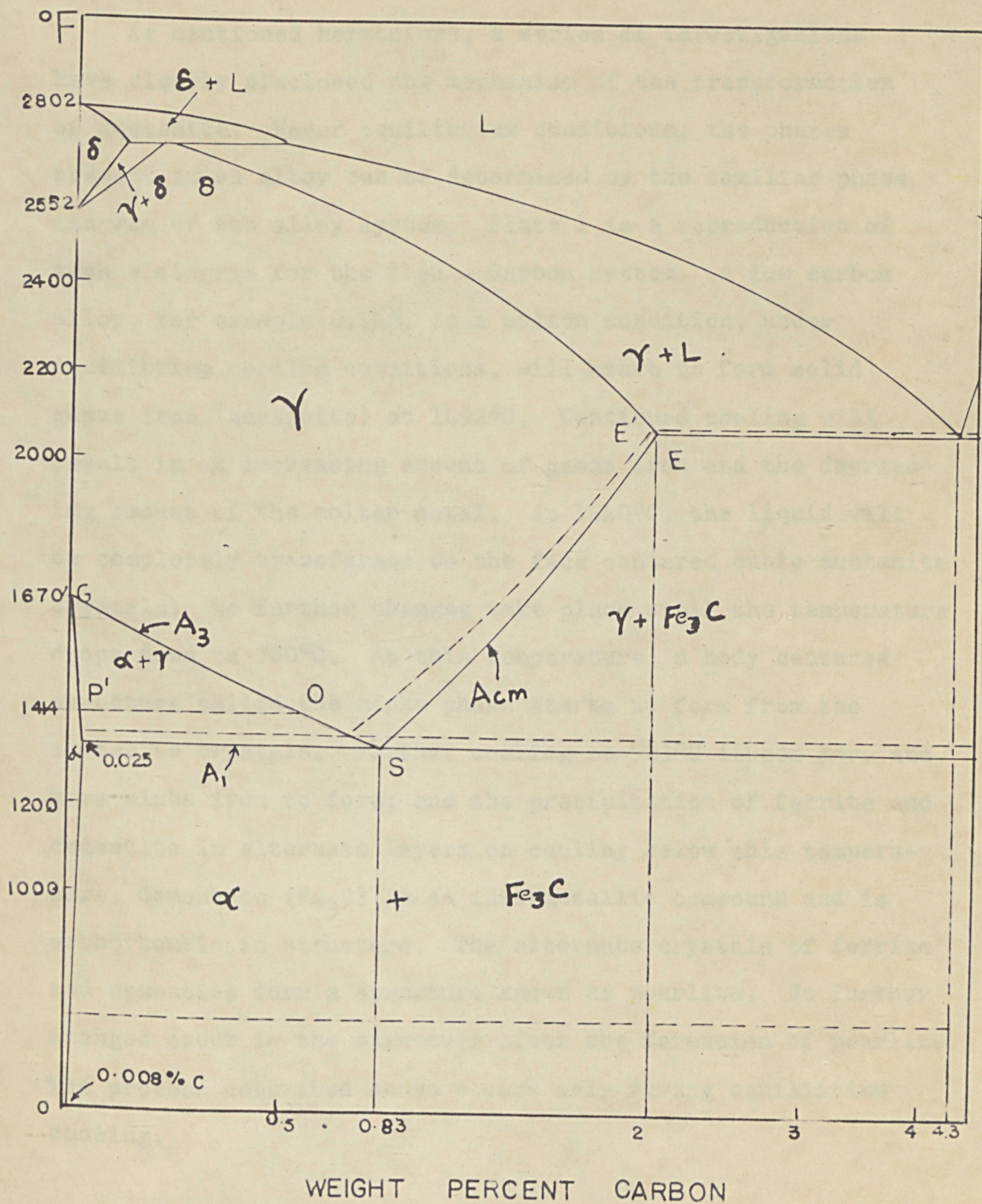
Of course, most of these beliefs were discarded in later years, but the process of hardening and tempering were still

not well understood. Quenching and tempering therefore remained an art.

The rational explanation of the process of subcritical transformation was not disclosed until recently. It dates back to 1929 when the actual and thorough investigation of the transformation of austenite was begun in the laboratories of the United States Steel Corporation in Kearney, New Jersey. This work served to remove the mysteries of the "hows" and "whys" the properties of steels are altered by chemical composition and heat-treatment. The most significant contribution of this line of work was the development of the fact that the properties of a given steel are functions of the heat treatment and microstructure.

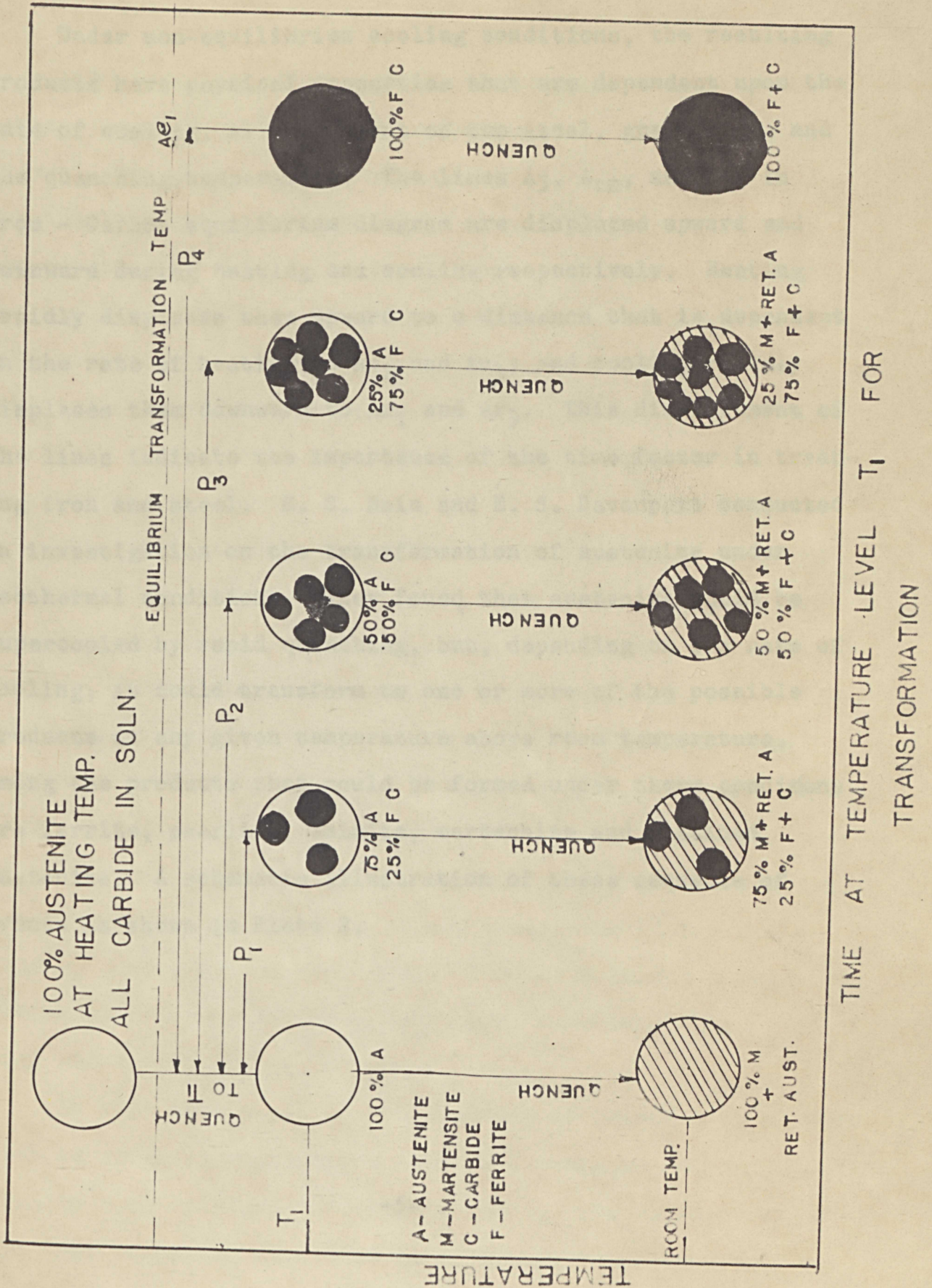
PLATE I

IRON-CARBIDE DIAGRAM



Equilibrium and Non-equilibrium Cooling

As mentioned heretofore, a series of investigations have clearly disclosed the mechanism of the transformation of austenite. Under equilibrium conditions, the phases present in an alloy can be determined by the familiar phase diagram of the alloy system. Plate 1 is a reproduction of such a diagram for the Iron - Carbon System. A low carbon alloy, for example 0.5%C, in a molten condition, under equilibrium cooling conditions, will start to form solid gamma iron (austenite) at 1492°C. Continued cooling will result in an increasing amount of gamma iron and the decreasing amount of the molten metal. At 1440°C, the liquid will be completely transformed to the face centered cubic austenite crystals. No further changes take place until the temperature drops down to 780°C. At this temperature, a body centered structure called the alpha phase starts to form from the austenite crystals. Further cooling to 723°C causes more and more alpha iron to form; and the precipitation of ferrite and cementite in alternate layers on cooling below this temperature. Cementite (Fe_3C) is an intermetallic compound and is orthorhombic in structure. The alternate crystals of ferrite and cementite form a structure known as pearlite. No further changes occur in the structure after the formation of pearlite. The process described above occurs only during equilibrium cooling.



Under non-equilibrium cooling conditions, the resulting products have physical properties that are dependent upon the rate of cooling, alloy content of the steel, grain size, and the quenching temperature. The lines A_3 , A_{cm} , and A_1 , in Iron - Carbon equilibrium diagram are displaced upward and downward during heating and cooling respectively. Heating rapidly displaces them upward to a distance that is dependent on the rate of heating to Ac_1 and Ac_3 ; and cooling rapidly displaces them downward to Ar_1 and Ar_3 . This displacement of the lines indicate the importance of the time factor in treating iron and steel. E. C. Bain and E. S. Davenport conducted an investigation on the transformation of austenite under isothermal conditions. They found that austenite could be supercooled by rapid quenching, but, depending on the rate of cooling, it could transform to one or more of the possible products at any given temperature above room temperature. Among the products that could be formed under these conditions are ferrite, pearlite, bainite, martensite and retained austenite. A schematic illustration of these sequence of events is shown in Plate 2.

Formation of Martensite and Retained Austenite

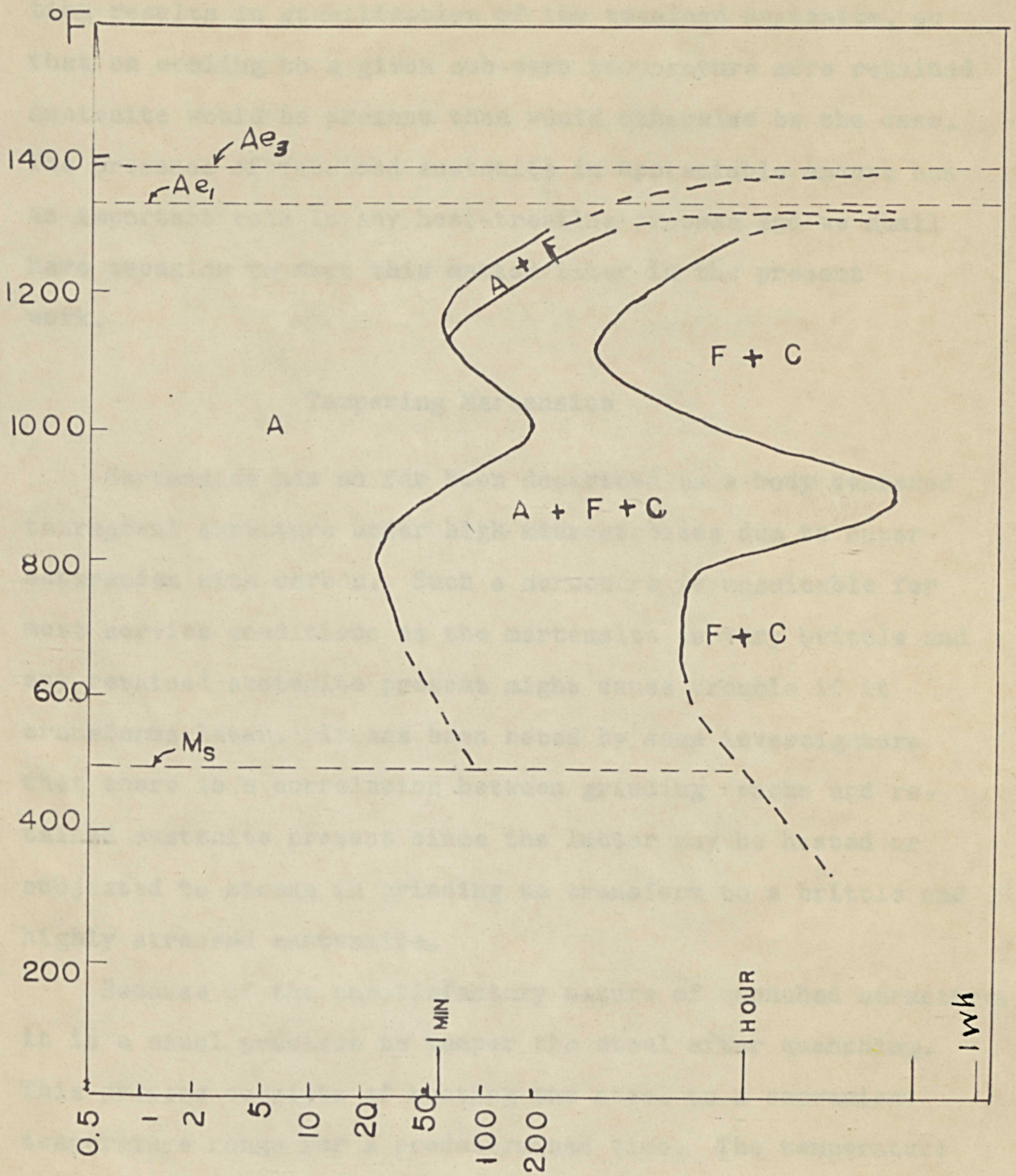
The formation of martensite differs markedly from the other products of austenite transformation. In a hardening quench, the desired transformation product is martensite and the cooling is made rapid enough to prevent austenite from decomposing to any of the other products. Fortunately, time is a major factor in the formation of these other products, and so their formation can be suppressed by fast cooling through the temperature range where they form. Thus it is possible to supercool austenite to the particular range where martensite will form.

The birth of martensite from the parent phase is quite an event. It starts forming at a temperature designated as M_s and stops at M_f . (A Time-Temperature-Transformation Curve for the steel under consideration is reproduced in Plate 3.) Further, it starts forming during cooling at M_s and stops when the cooling is stopped. Martensite forms from the parent phase by the process of nucleation and shear.. More and more of these shears take place during cooling and so the reaction proceeds primarily by birth of new martensite plates rather than by growth of previous ones. The newly formed martensite plates have a body centered tetragonal structure that is under high microstresses.

In most steels, it is most unusual to obtain a structure that is entirely martensitic at room temperature -- there is usually some retained austenite present. The elimination of the last traces of austenite is difficult to achieve even by

PLATE 3

I - T DIAGRAM OF X-9165



refrigeration. Holding at room temperature before refrigeration results in stabilization of the retained austenite, so that on cooling to a given sub-zero temperature more retained austenite would be present than would otherwise be the case. The presence of retained austenite in appreciable amount has an important role in any heat-treating process and we shall have occasion to meet this matter later in the present work.

Tempering Martensite

Martensite has so far been described as a body centered tetragonal structure under high microstresses due to supersaturation with carbon. Such a structure is unsuitable for most service conditions as the martensite is very brittle and any retained austenite present might cause trouble if it transforms later. It has been noted by some investigators that there is a correlation between grinding cracks and retained austenite present since the latter may be heated or subjected to stress in grinding to transform to a brittle and highly stressed martensite.

Because of the unsatisfactory nature of quenched structures, it is a usual practice to temper the steel after quenching. This process consists of heating the steel to a convenient temperature range for a predetermined time. The temperature range for tempering is below the critical transformation range.

Various investigators have established that as the temperature and time of tempering are increased, the whole process passes through four stages namely:

- (1) Decomposition of tetragonal martensite which takes place on heating up to 400°F.
- (2) Decomposition of retained austenite which takes place from 400°F to 600°F.
- (3) Formation of Cementite at about 600°F to 1000°F.
- (4) The fourth stage is the formation of alloy carbides and this occurs at higher temperatures (above 1000°F)

Techniques Used to Study Tempering Behavior

Tempering has been described as heating a martensite structure to higher temperatures to produce the desired properties. Investigations are usually based on:

- (a) X-ray diffraction changes in each cycle of tempering.
- (b) Magnetic and Dilation changes.
- (c) Volume changes.
- (d) Hardness changes.
- (e) Changes in Microstructure.

Most of these methods are used to supplement and confirm the data obtained from the other. Studies based on hardness and microstructure have been used more frequently. This is perhaps because indentation hardness is the simplest measure of a tempered structure or the "degree of tempering" while studies of the microstructures enable one to follow the internal changes that take place as the tempering progresses.

Undoubtedly, the hardness method is not an exact method and in some cases (as when temper brittleness occurs) pieces of the same quenched and tempered steel may have widely different properties at the same hardness. However, hardness measurement has the advantage of being simple. It is on the changes in hardness that accompany tempering at given temperatures and times that the present study is based.

PART II: THE INVESTIGATION

Apparatus and Materials

It was decided that the tempering studies will be done by the hardness method with measurements being made on the Rockwell hardness testing machine since the facility for this type of experimental work is available in the Metallurgy Department of Montana School of Mines. It is apparent that furnaces are needed, preferably electric furnaces for ease of temperature control. One of the furnaces would serve for preliminary heating to render the steel austenitic, and the other furnaces maintaining constant temperature baths for tempering of the martensite formed by quenching. Besides the furnaces, the procedure calls for temperature measuring devices for the constant temperature baths, pots to hold the baths, mechanical devices to handle the hot specimens without touching them with cold tongs, and finally, an oil quenching bath to cool the specimens to room temperature.

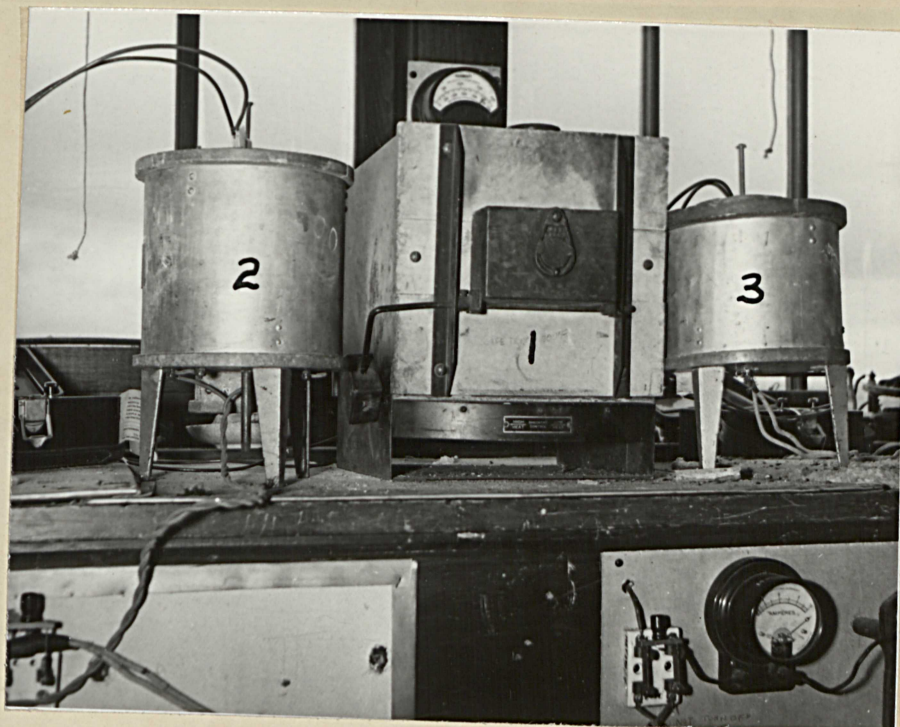
The heating furnace is of the muffle type equipped with temperature measuring devices. The temperature can be read on a meter on top of the furnace.

The quenching bath furnaces are cylindrical multiple unit electric resistance furnaces. The maximum temperatures obtainable from them are 1800°F. They were, however, not used above 1200°F. Two of these furnaces were used to speed

up the investigation. The quenching medium in one pot is lead, and a low melting point alloy of lead, bismuth and tin in the other. Temperature measurements in the bath furnaces were made with chromel-alumel thermocouples which were accurate to within $\pm 5^{\circ}\text{F}$.

The mechanical aids in handling specimens consisted of ten inch lengths of nickel wires which were attached securely to one end of each specimen. The heating furnace and the pot furnaces were conveniently handled by cupel tongs to avoid burns to the user.

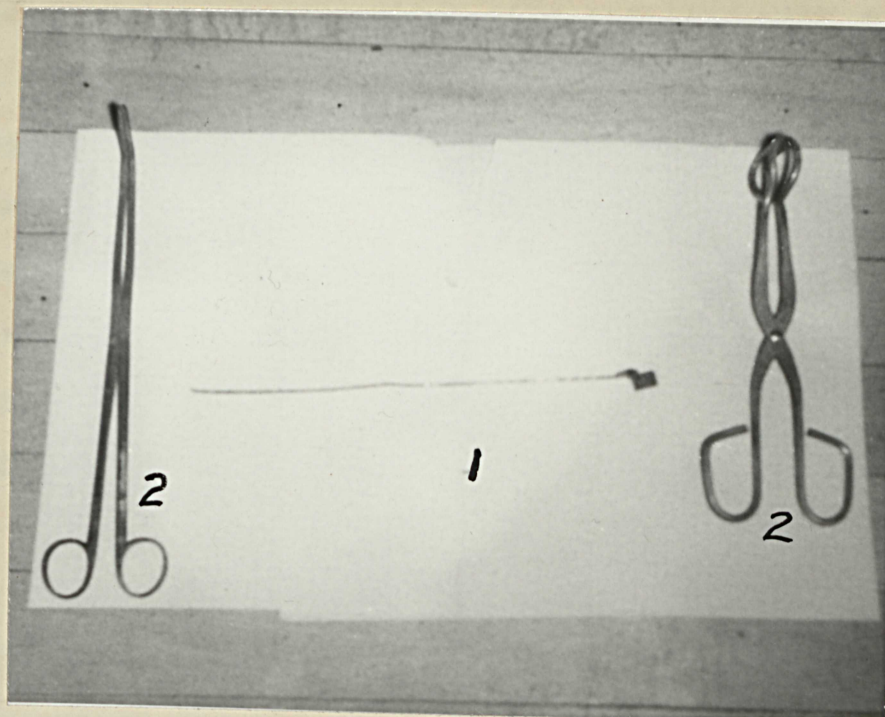
The room temperature quenching medium was oil and it was contained in a can placed conveniently near the furnaces. The whole set-up was assembled very close together so that no time was lost in transferring the specimens from one furnace to another. Plate 4 shows the arrangement of the furnaces for the investigations.



FURNACE ARRANGEMENT

1. AUSTENITIZING FURNACE
2. TEMPERING FURNACE LEAD BATH
3. TEMPERING FURNACE LOW MELTING-
POINT ALLOY

PLATE 4 b



1. SPECIMEN AND HOLDER
2. CUPEL TONGS FOR FURNACE HANDLING

Laboratory Procedure

Preliminary work on the steel consisted of cutting specimens into a convenient size sections with a power saw. The steel as supplied by the manufacturer came in four foot lengths of $\frac{1}{4}$ by $\frac{1}{4}$ inch cross-section. For this investigation, they were cut up in lengths of $\frac{1}{2}$ inch thus giving the dimension of each specimen as $\frac{1}{4}$ by $\frac{1}{4}$ by $\frac{1}{2}$ inch. This size is large enough for convenient handling and small enough to attain the bath temperatures quickly. One hundred and six such specimens were used for this study.

Metallurgical data was available for this steel from the manufacturer and the composition is listed in the introductory section of this paper. The proper austenitizing temperature for the steel as given by the manufacturer is 1650°F for 15 minutes. However, different temperatures and times were used in the present work as will be explained shortly.

Preliminary experiments with the steel consisted of austenitizing at 1650°F for 15 minutes and quenching in water at 20°C. The structure obtained was examined and was found to be mostly martensite. Other tests were made at 1750°F for the same time interval and the structure was again found to be completely martensite. It was also observed that the steel developed quenching cracks during the hardening quench in water. It was therefore decided that the steel was sensitive quench cracking and a less drastic quench (oil) should be used.

Austenitizing Cycles

The investigation was divided into two parts. In the first part, the austenitizing temperatures were varied. In this part, a set of thirty specimens was austenitized at temperatures of 1500°F, 1750°F, and 2000°F respectively, for thirty minutes and then quenched in oil at room temperature. This operation was necessary to study the effect of the austenitizing temperature in the subsequent tempering operation.

In the second part, an arresting quench was used in order to see what effect it would have on the tempering behavior in this steel. Four sets, of four specimens each, were austenitized at 2000°F for thirty minutes and the quenching was arrested at four different temperatures above the M_s point of the steel before quenching to room temperature in oil. This later process was closely correlated with the S-curve as determined by Dilip Das and reproduced here in Plate 3, to determine the holding time limit at the different temperatures. The arresting temperatures used were 1200°F, 1000°F, 800°F, and 600°F; and the time of holding at these temperatures was fifteen seconds. In this time interval, the steel is still in the unstable austenite condition.

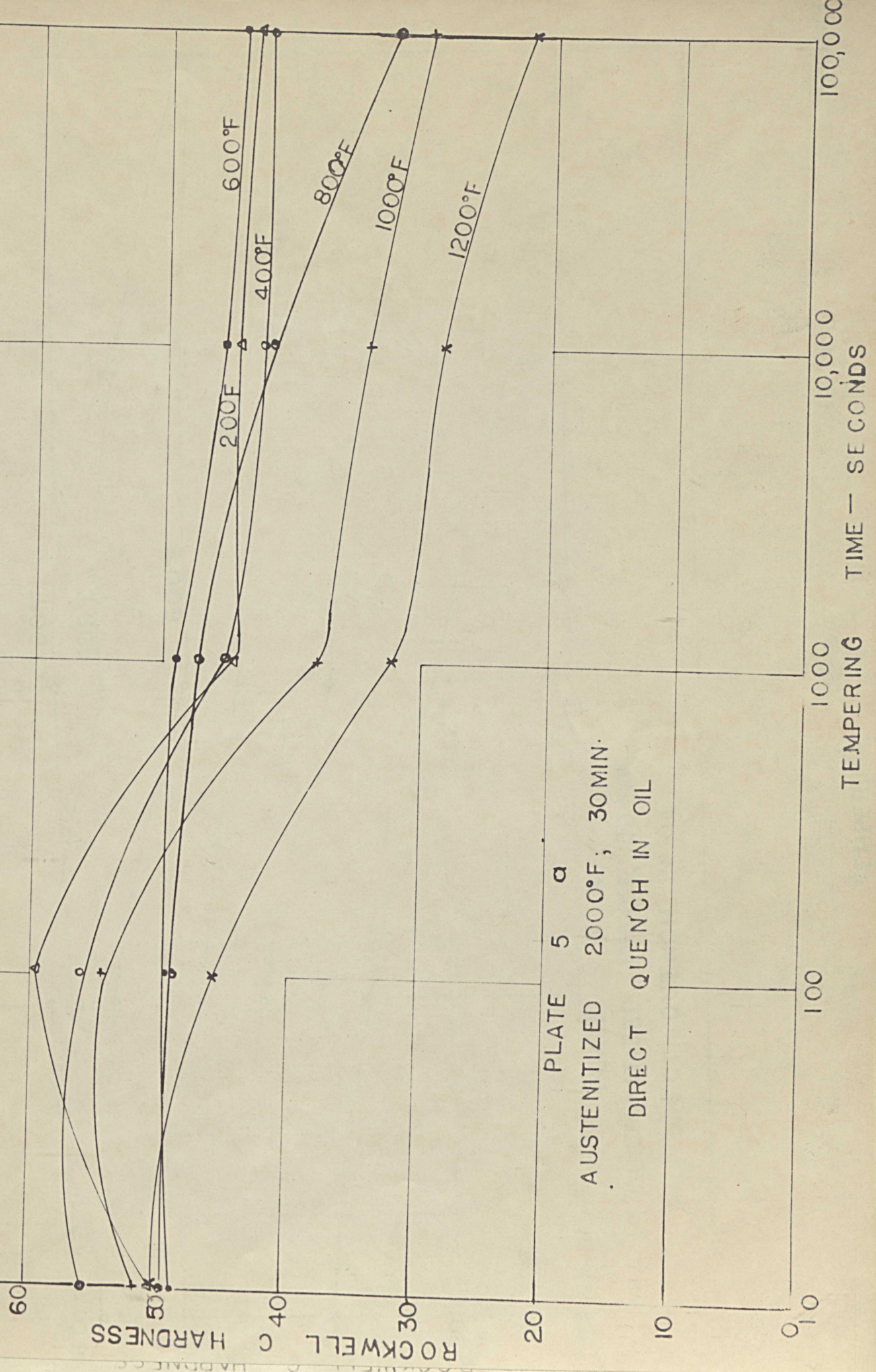
Tempering Cycles

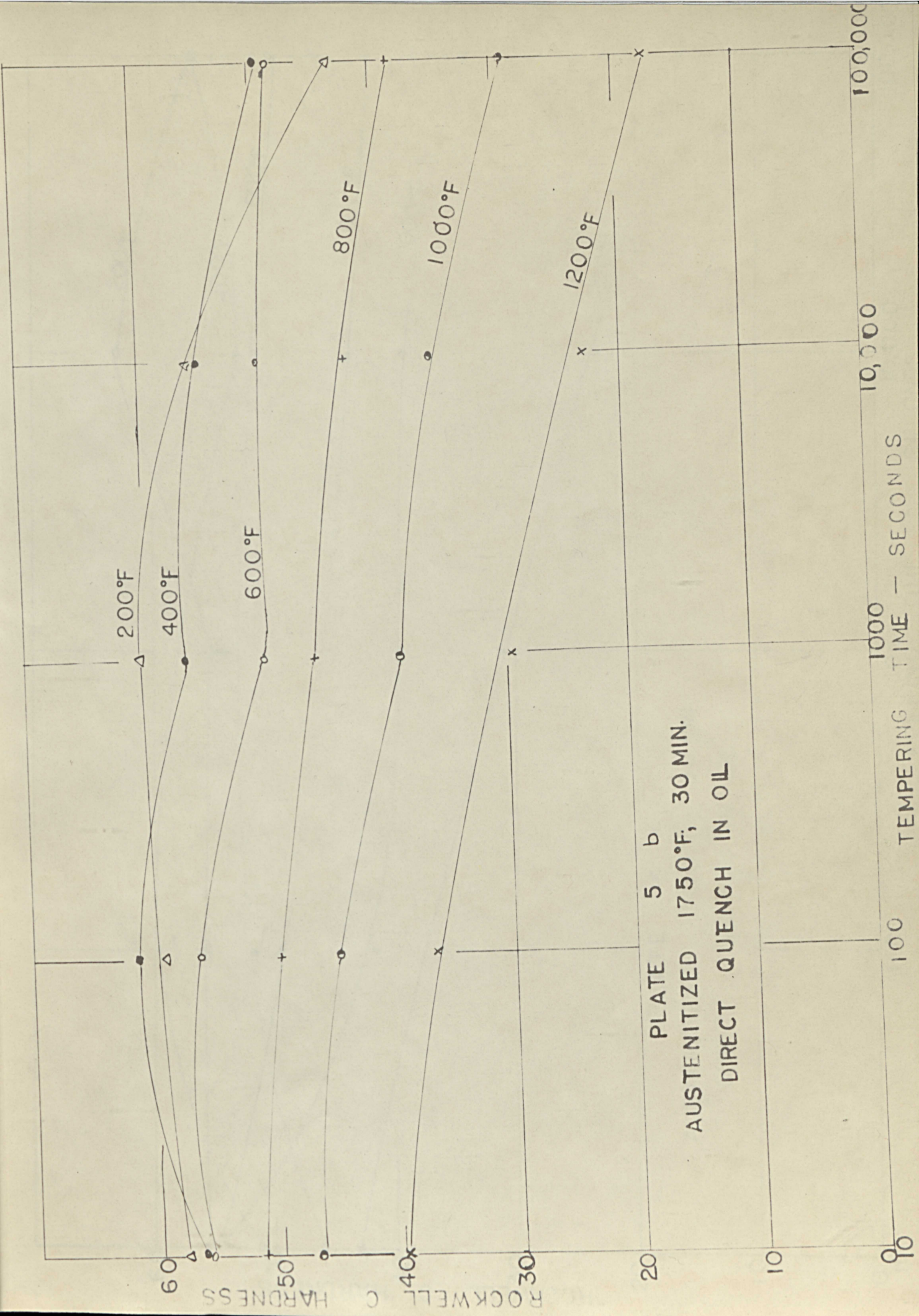
The first series of tempering studies were done on the direct quench specimens. The specimens from each of the three austenitizing temperatures were tempered at 200°F, 400°F, 600°F, 800°F, 1000°F, and 1200°F; for lengths of times ranging from 10 seconds to 100,000 seconds (27+ hours). Single tempering was used with each specimen tempered only once at each temperature and time. Quenching after tempering was done in water at room temperature. Hardness measurements were made across the length of the tempered specimens. About ten such hardness readings were made on two, and sometimes three, faces of the carefully ground steel and the average was taken. Table I (a), (b), and (c) shows the data for this set of work for the three austenitizing temperatures.

In part two of this work, the arrested quench specimens were also tempered at the arresting temperatures (i.e. 1200°F, 1000°F, 800°F, and 600°F) for time intervals ranging from 10 seconds to 10,000 seconds (23/4 hours). Cumulative single, double, tertiary and quaternary tempering were used on each specimen at each tempering temperature. By this is meant that at each tempering temperature, the hardness was taken after a short tempering time, then the same specimen was tempered again for the next longer time, and the hardness again was taken; and so on until the maximum time is reached. This series of oper-

ations was necessary in order to evaluate quantitatively the tempering process in this steel. Table 2 (a), (b), (c), and (d) shows the data on the hardness values at each tempering temperature and time.

All tempering operations were carried out in the bath furnaces except the 200°F tempering where boiling water was used.





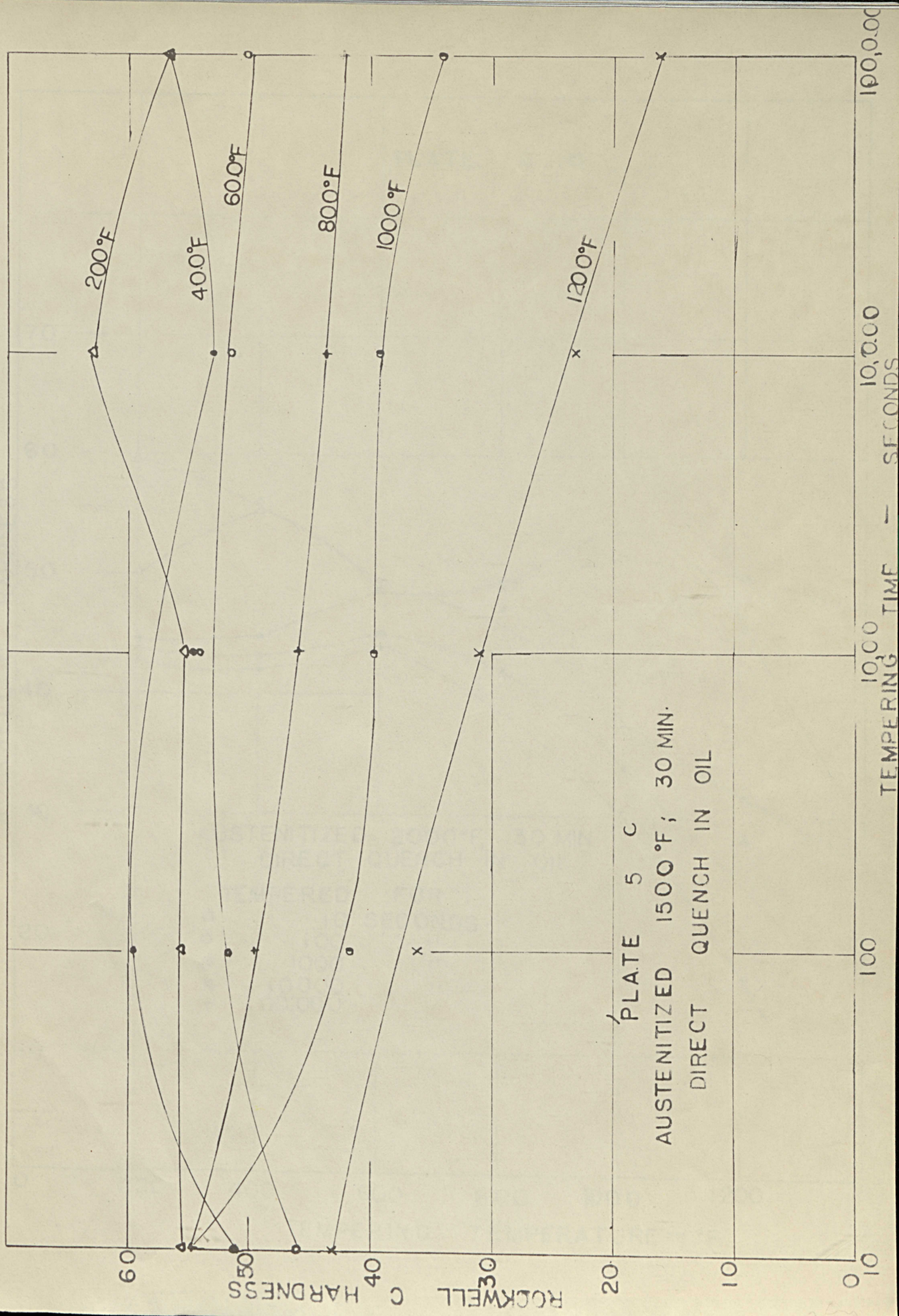


PLATE 6 a

ROCKWELL C HARDNESS

AUSTENITIZED 2000°F, 30 MIN
DIRECT QUENCH IN OIL

TEMPERED FOR ;
10 SECONDS

Δ 100
 ○ 1000
 ● 10000
 x 100,000
 + 1,000,000

TEMPERING TEMPERATURE — °F

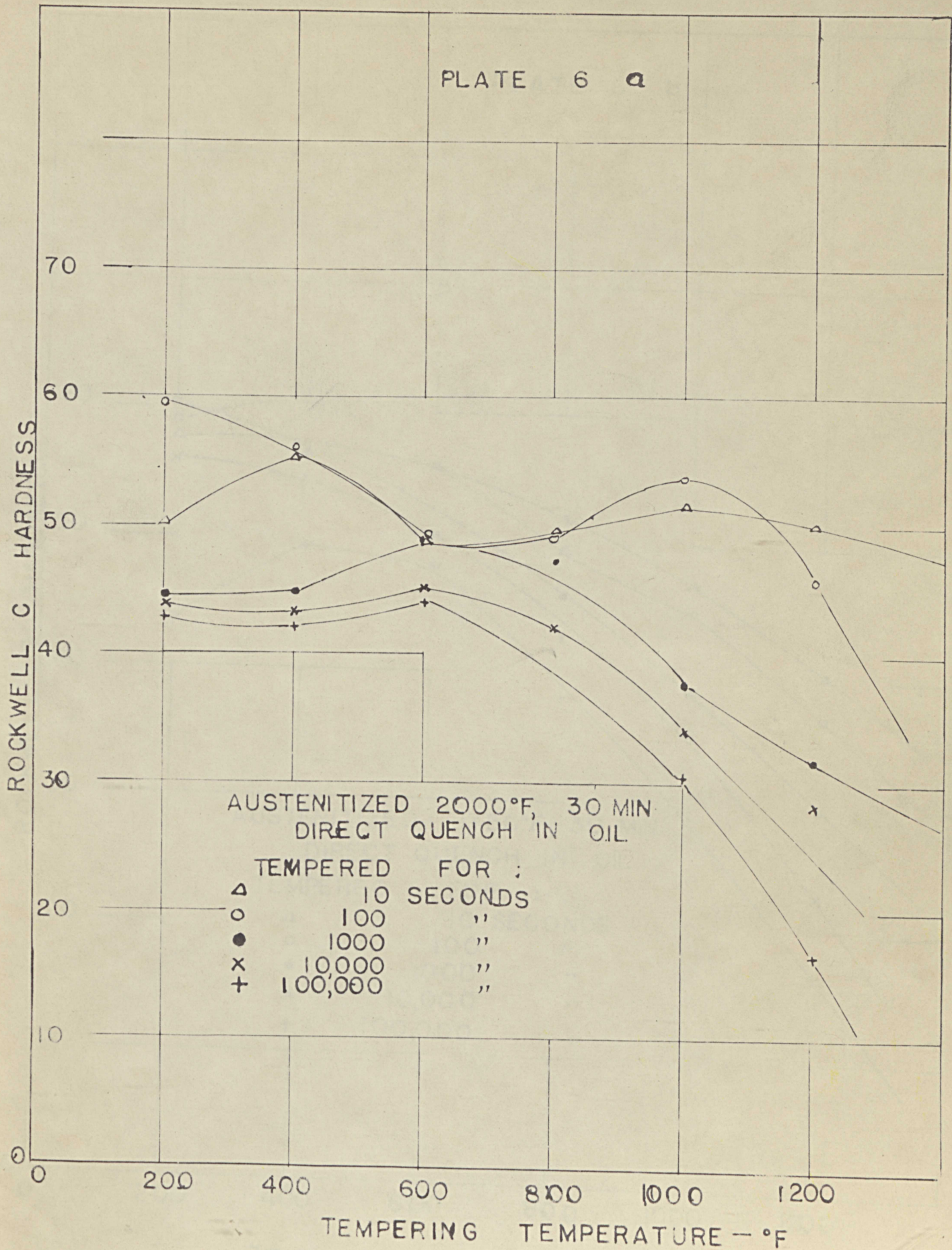


PLATE 6 b

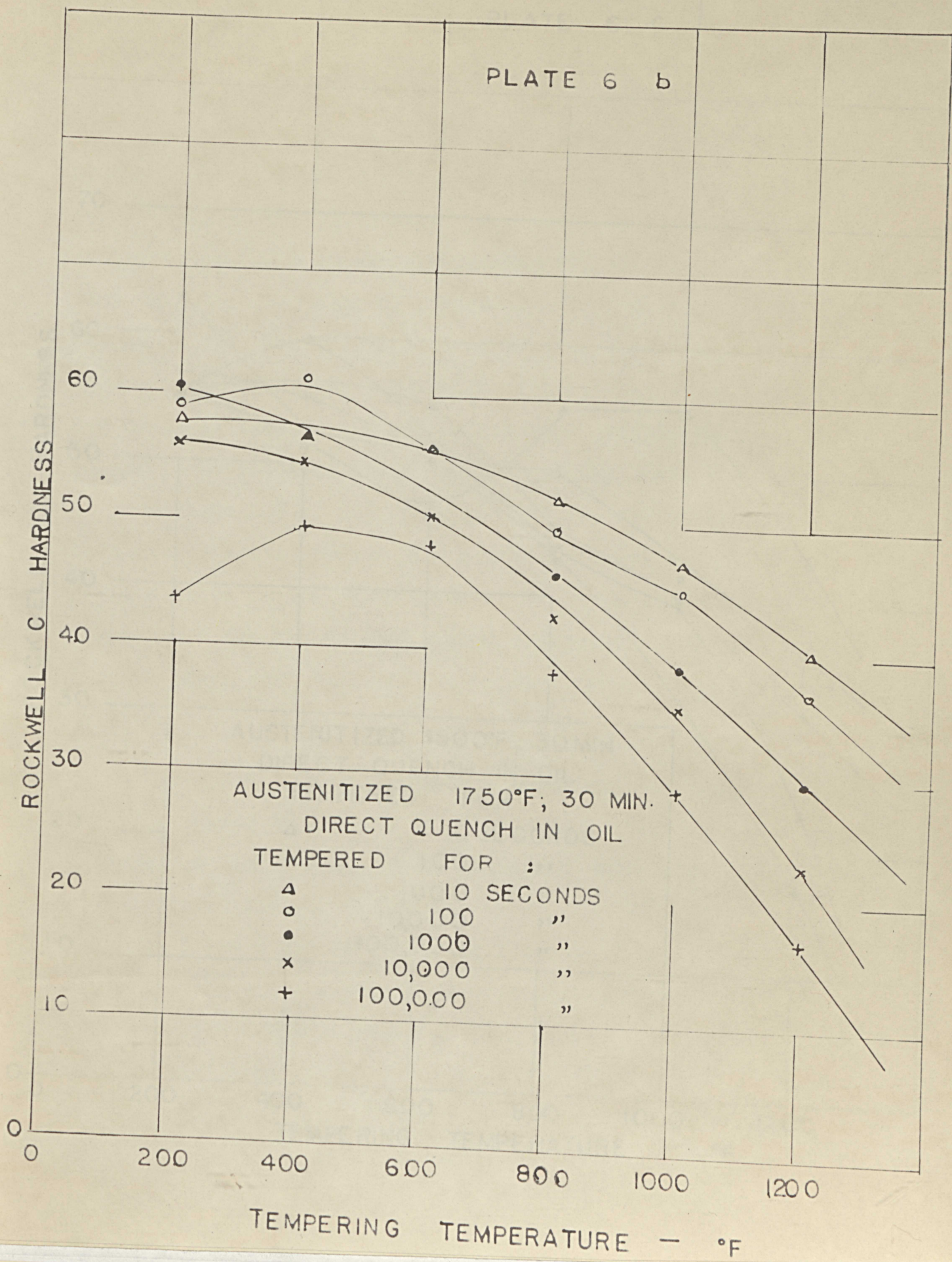
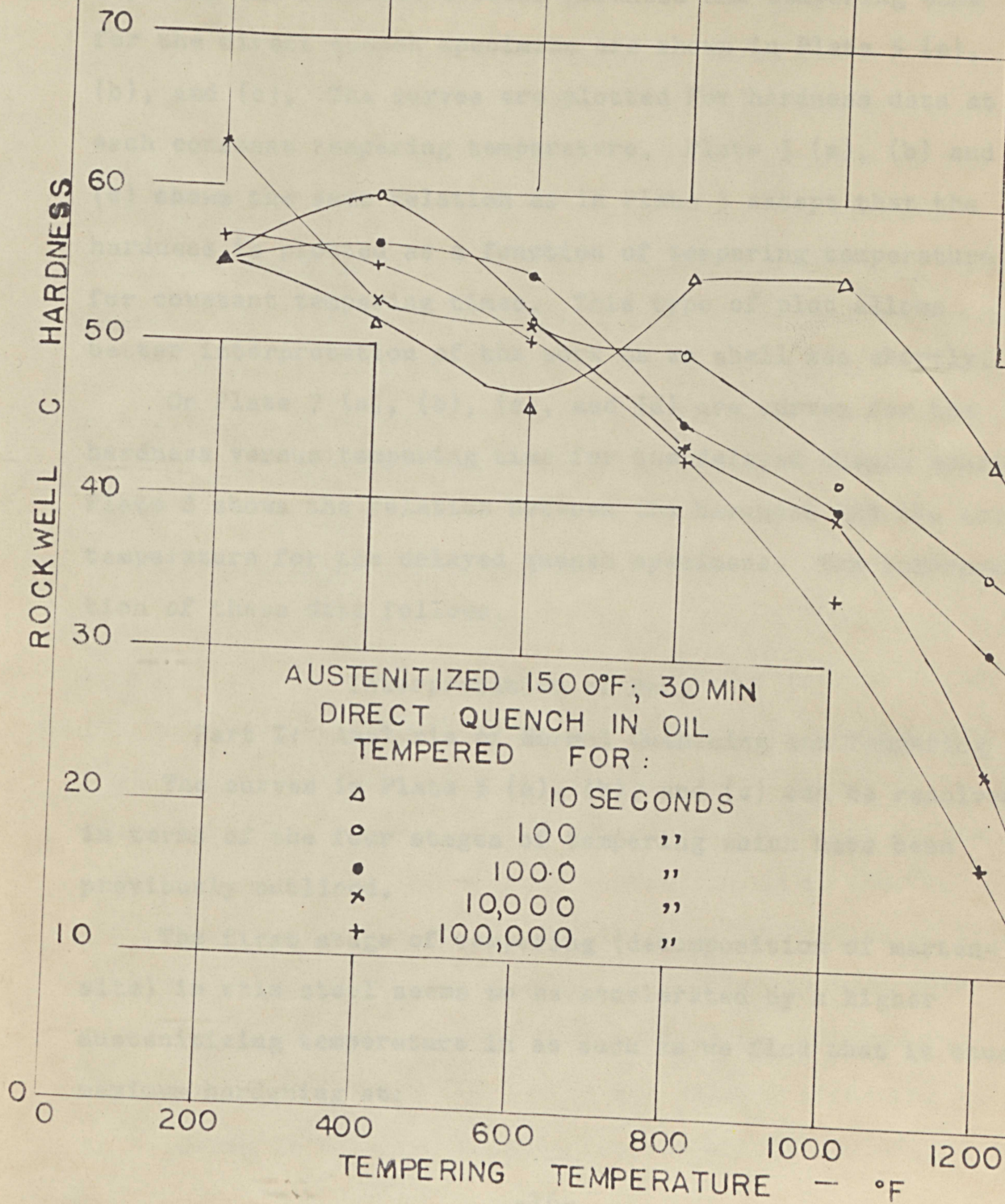


PLATE 6 c

ROCKWELL C HARDNESS

AUSTENITIZED 1500°F; 30 MIN
DIRECT QUENCH IN OIL
TEMPERED FOR:

Δ	10 SECONDS
○	100 "
●	100.0 "
×	10,000 "
+	100,000 "



PART III. DATA AND RESULTS

The hardness data are shown in tables 1 and 2. Curves depicting the relation between hardness and tempering time for the direct quench specimens are shown in Plate 5 (a), (b), and (c). The curves are plotted for hardness data at each constant tempering temperature. Plate 5 (a), (b) and (c) shows the same relation as in Plate 5 except that the hardness is plotted as a function of tempering temperature for constant tempering times. This type of plot allows better interpretation of the work as we shall see shortly.

On Plate 7 (a), (b), (c), and (d) are curves for the hardness versus tempering time for the delayed quench specimens. Plate 8 shows the relation between the hardness and the holding temperature for the delayed quench specimens. The interpretation of these data follows.

Interpretation of Data

Part I: Analysis of Normal Quenching and Tempering

The curves in Plate 5 (a), (b), and (c) can be resolved in terms of the four stages of tempering which have been previously outlined.

The first stage of tempering (decomposition of martensite) in this steel seems to be accelerated by a higher austenitizing temperature in as much as we find that it caused maximum hardening at:

100 seconds following austenitization at 2000°F.

1000 seconds following austenitization at 1750°F.

10,000 seconds following austenitization at 1500°F.

upon tempering at 200°F. At 400°F the distinction is less prominent but seems to follow the same pattern.

The second state of tempering (decomposition of retained austenite) is indicated by the increase in hardness in the 600°F curves. Here again, the decomposition of retained austenite seems to be delayed by a lower austenitizing temperatures. The decomposition at 600°F after austenitizing at 2000°F does not cause the marked change in hardness found after the austenitizing treatment, but this is probably due to the fact that it proceeds very rapidly at this temperature and the major effect is missed.

The softening due to the third stage (formation of cementite) likewise proceeds more rapidly with higher austenitizing temperature so that a rapid softening which occurs between 10,000 and 100,000 seconds at 800°F after austenitizing at 2000°F does not appear after austenitizing at the other two temperatures and seems to be the reaction which is completing itself between 10 and 100 seconds at 1000°F after austenitizing at 1500°F.

The commencement of the fourth stage (formation of alloy carbides), while not occurring at an earlier time seems to have greater effect with increasing austenitizing temperature. This is shown in the curves for 1000°F and delayed softening is also in evidence in the 1200°F curve for the sample austenitized at

2000°F. This is not evident in the analogous curves for the other two austenitizing temperatures and is probably due to the fact that some of the more refractory alloy carbides were not dissolved during the austenitizing treatment.

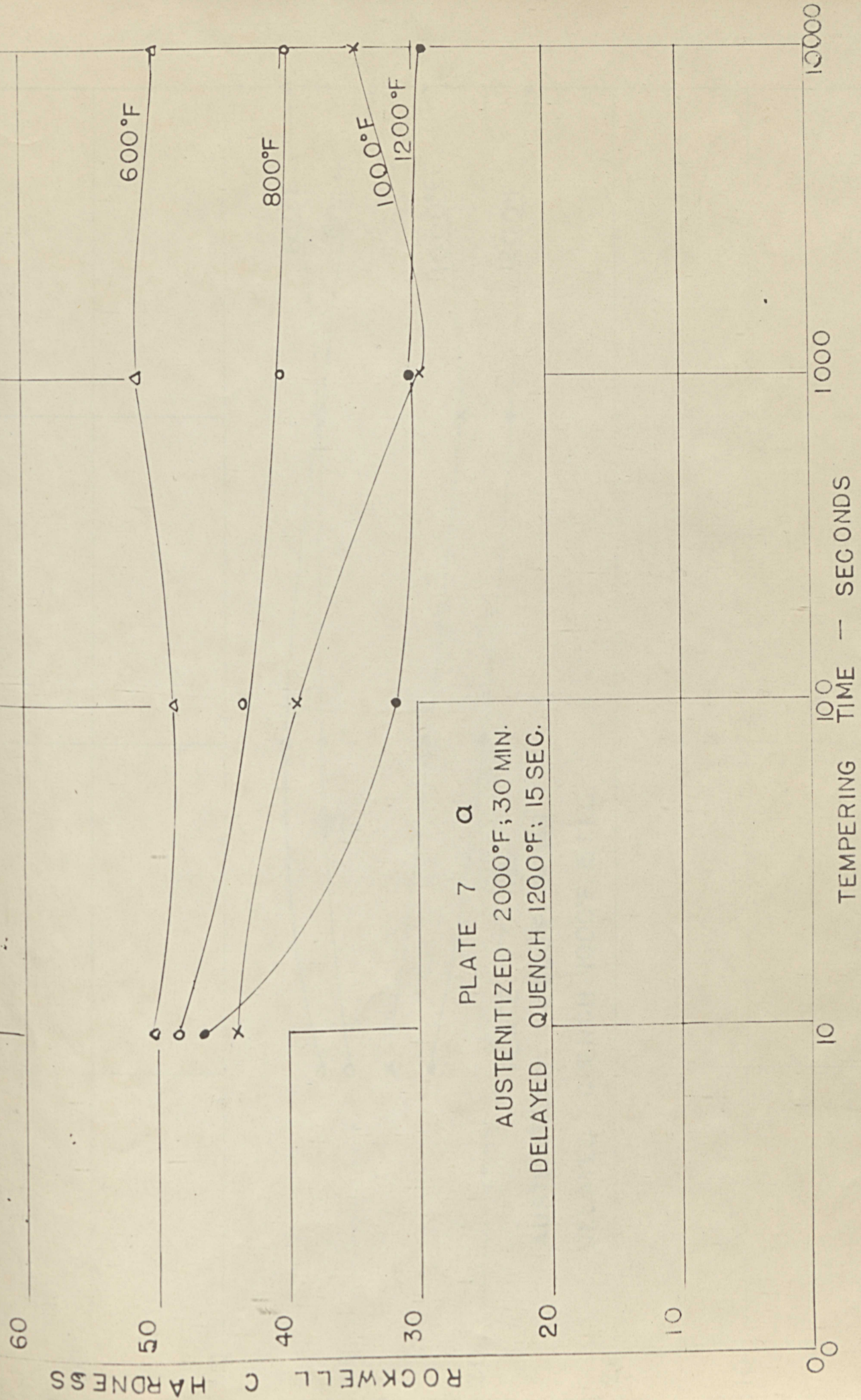
Part 2: Effect of Delay in Quenching

The fifteen seconds delay at the various temperatures after austenitizing at 2000°F is shown in the curves on Plates 8 (a), (b), (c), and (d) plotted to show the change in hardness with holding temperatures at tempering times of 10 100 seconds, 1000 seconds and 10,000 seconds. The first two of these show a general downward trend in hardness with holding temperature while the curves at 10,000 seconds show a gradual upward trend in all cases.

What this seems to indicate is that the delay in quenching at all temperatures first conditions the martensite so that softening is much more rapid in the first 1000 seconds. That is, at any tempering temperature, the hardness is less following the holding treatment than with no hold if we are using tempering times less than 1000 seconds.

When we hold for 15 seconds we find that for a given tempering temperature, the holding causes an increase in hardness at all temperatures above the 1000 seconds tempering time.

The explanation of this seems to lie in the fact that the holding results in the formation of nuclei of cementite in the austenite. Thus the rapid softening at shorter times would



60

50

40

30

20

10

00

ROCKWELL C HARDNESS

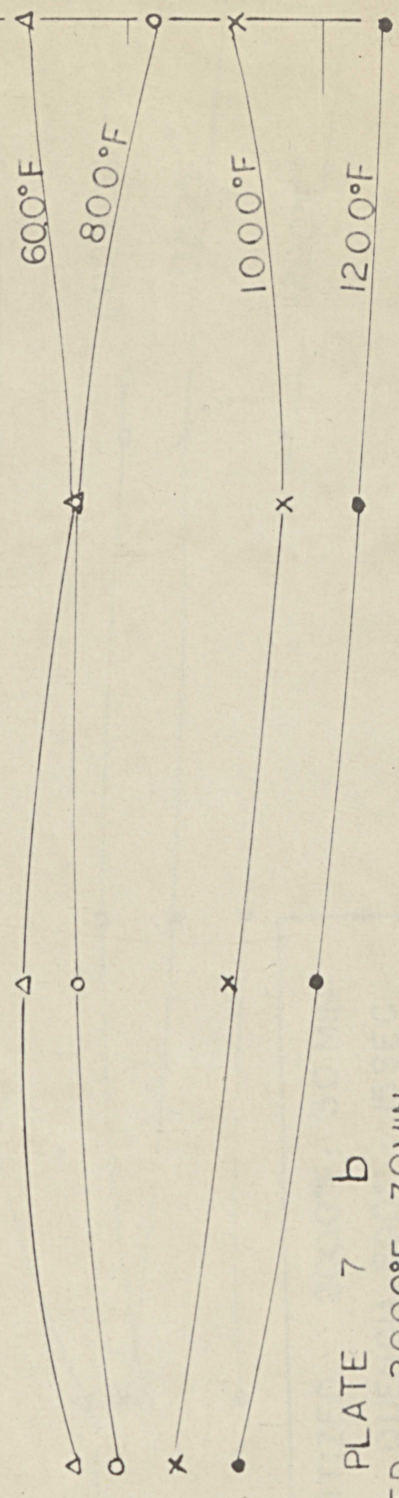


PLATE 7 b

AUSTENITIZED 2000°F, 30 MIN.

DELAYED QUENCH 1000°F, 15 SEC.

TEMPERING TIME — SECONDS.

1000

10000

PLATE 7 C

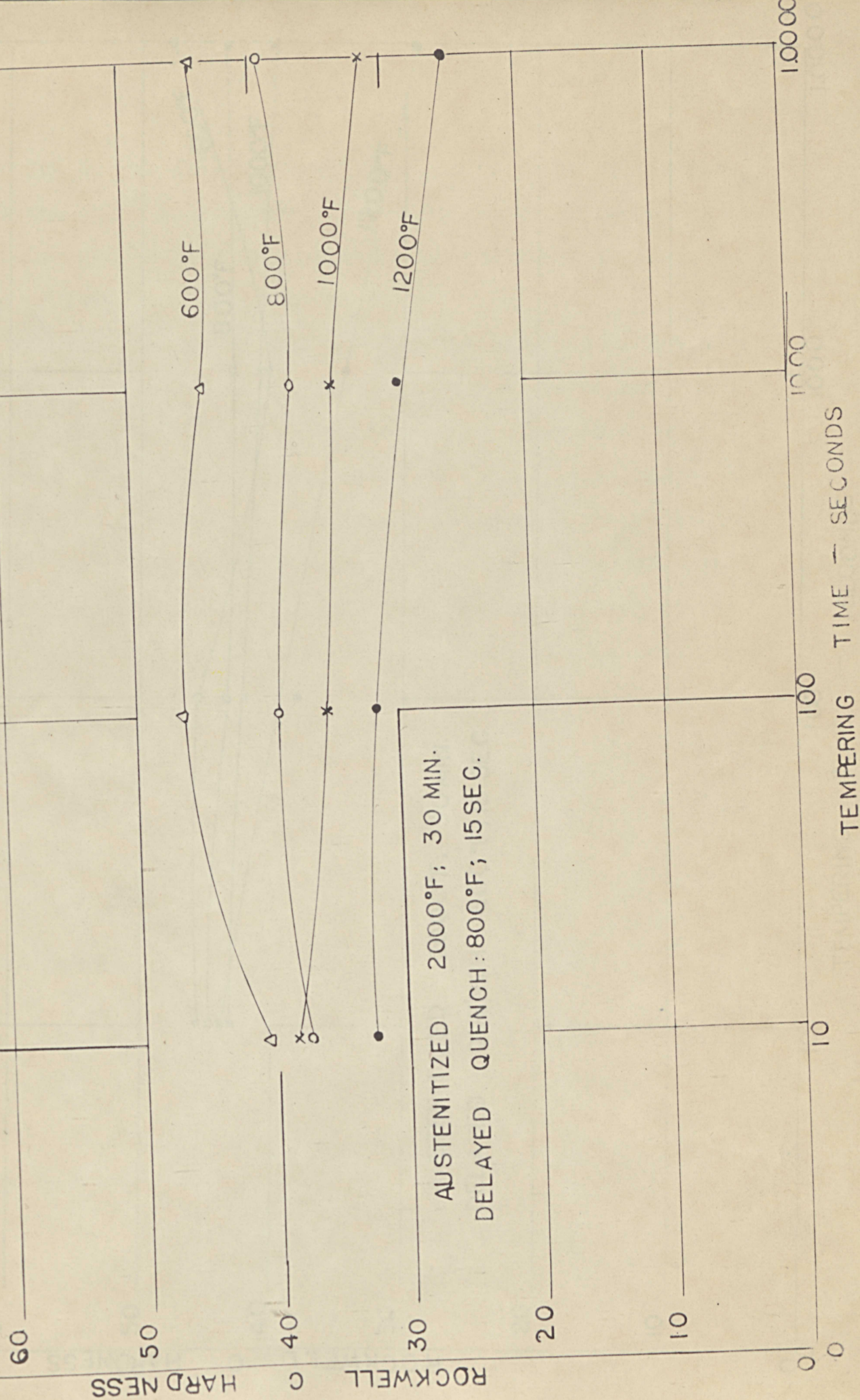


PLATE 7 d

60

50

40

30

20

10

0

ROCKWELL C HARDNESS

AUSTENITIZED 2000°F; 30 MIN.
 DELAYED QUENCH 600°F; 15 SEC.

600°F

800°F

1000°F

1200°F

10

100

1000

10000

TEMPERING TIME — SECONDS



occur due to the lack of delay in the nucleation of cementite, while the delay in softening is due to growth of these cementite particles and their slow solution when the alloy carbides (fourth stage of tempering) are formed.

Another phenomena observed from these curves is the marked decrease in hardness at all tempering temperatures and times after holding at 800°F. Such a phenomena seems to be very peculiar. The writer believes this to be due to a greater conditioning of the austenite at this holding temperature with the result that more nuclei of cementite result in the austenite matrix. Softening of such a structure after tempering at various temperatures and for various times can therefore be attributed to a more rapid formation of cementite.

DATA AND RESULTS

Part 1. Direct Quench Studies

Table 1 (a)

Specimens Austenitized at 2000°F for 30 minutes

Rockwell "C" Hardness Obtained by Tempering at the Indicated Temperature and Time						
Time at Tempering	200°F	400°F	600°F	800°F	1000°F	1200°F
10 sec.	50.1	55.5	48.7	49.6	51.7	50.0
100 "	59.5	56.1	49.2	49.0	54.3	45.9
1000 "	44.5	44.9	49.9	47.1	37.8	32.0
10,000 "	44.1	42.6	45.3	42.2	34.0	28.3
100,000 "	43.0	42.0	44.7	32.3	30.6	21.4

Table 1 (b)

Specimens Austenitized at 1750°F for 30 minutes

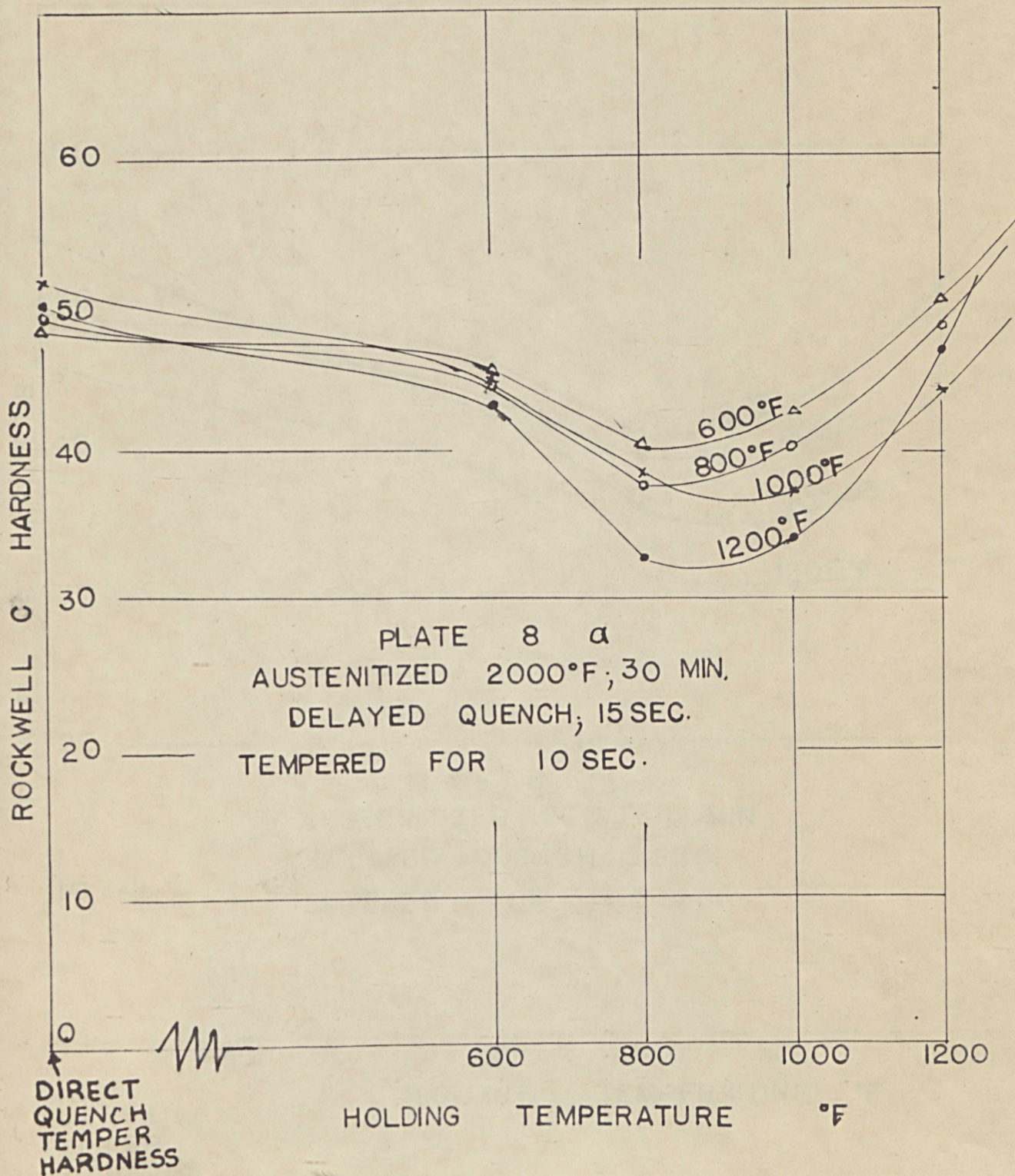
Rockwell "C" Hardness Obtained by Tempering at the Indicated Temperature and Time						
Time at Tempering	200°F	400°F	600°F	800°F	1000°F	1200°F
10 sec.	58.0	56.6	56.0	51.8	47.0	39.9
100 "	59.0	61.4	56.0	49.7	44.7	36.7
1000 "	60.0	56.7	50.0	46.0	38.8	29.7
10,000 "	56.0	55.2	50.0	42.6	35.6	23.0
100,000 "	43.5	49.6	48.3	38.3	29.0	17.4

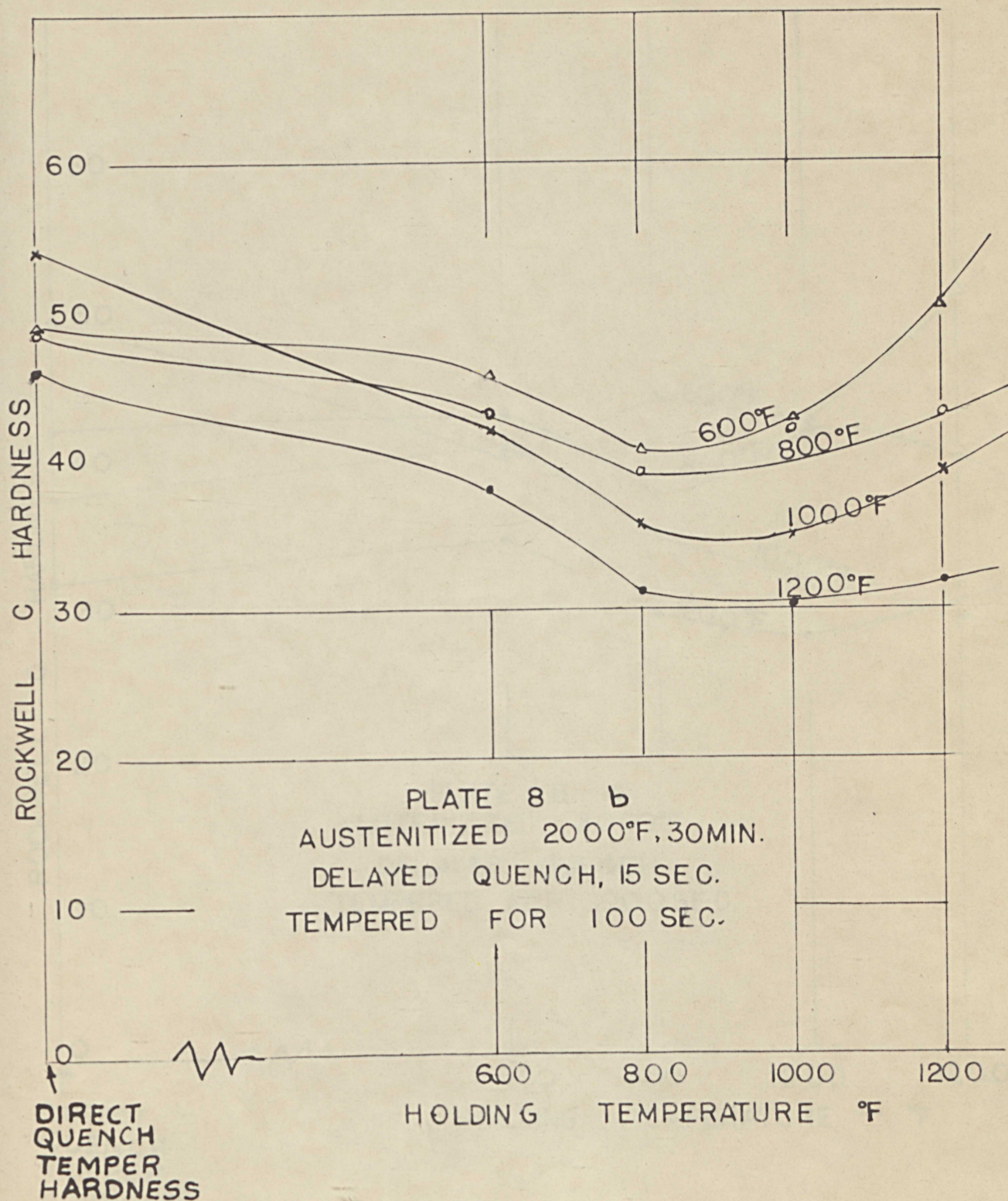
Table 1 (c)

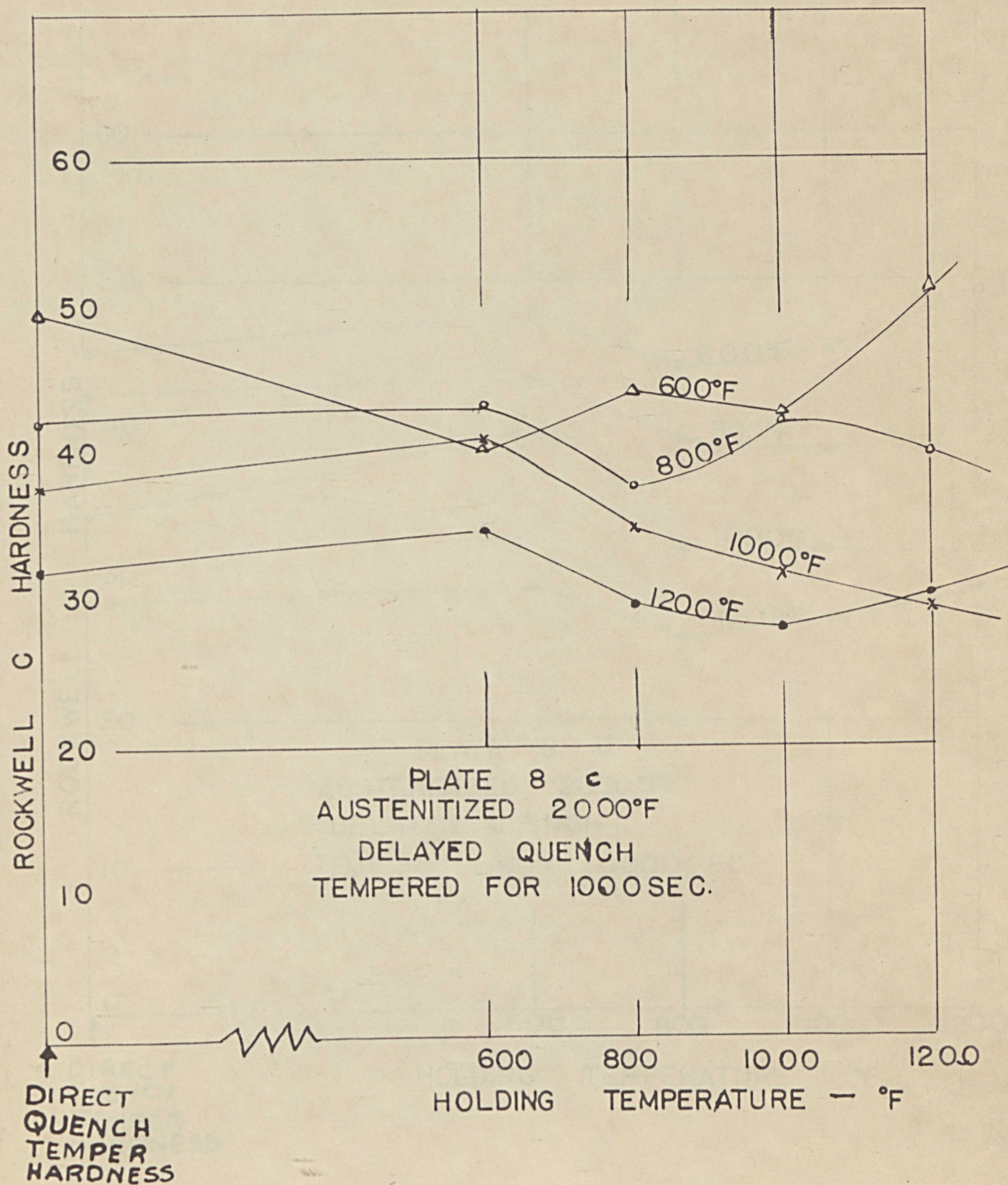
Specimens Austenitized at 1500°F for 30 minutes

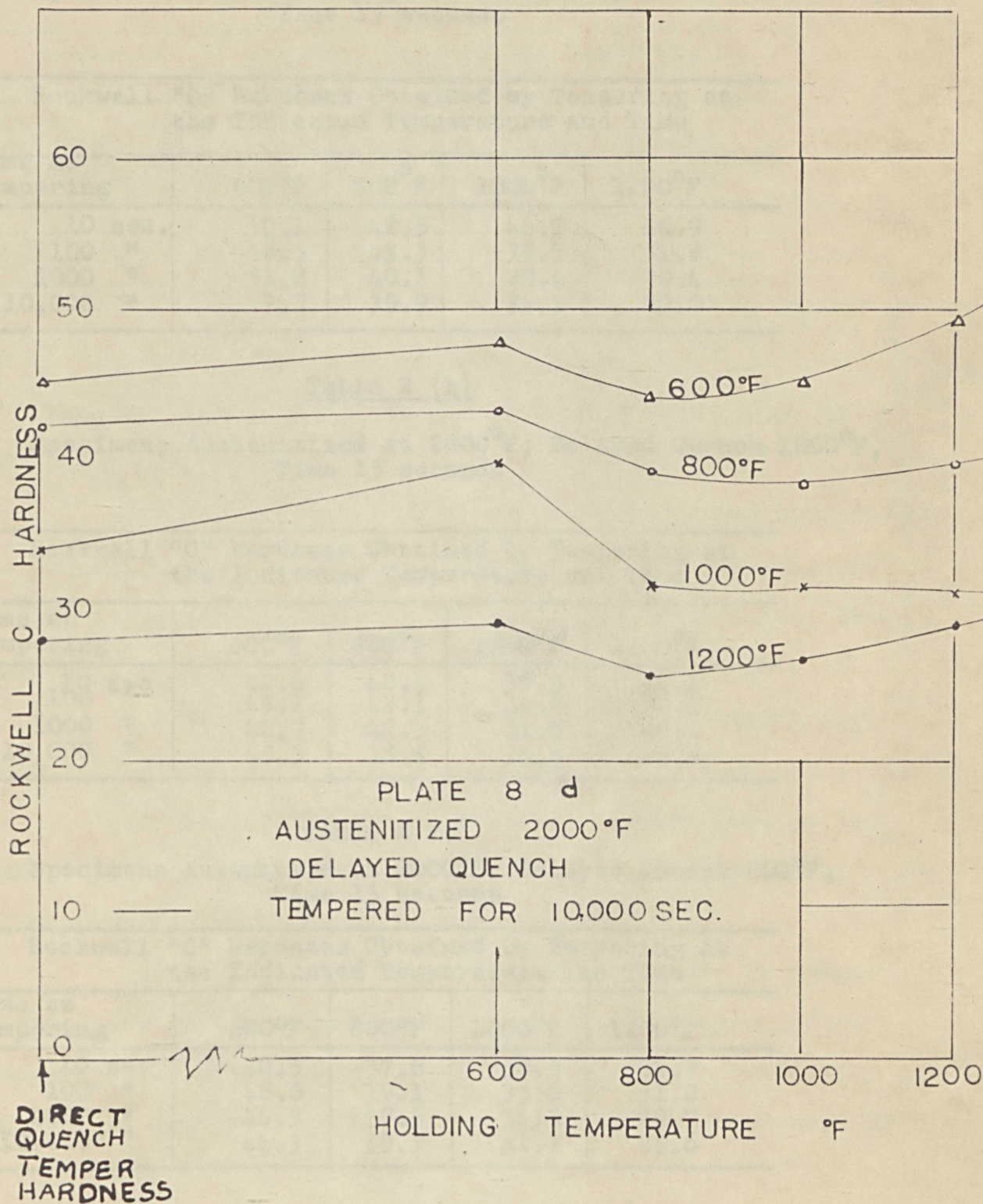
Rockwell "C" Hardness Obtained by Tempering at the Indicated Temperature and Time						
Time at Tempering	200°F	400°F	600°F	800°F	1000°F	1200°F
10 sec.	55.4	51.3	46.0	55.0	54.8	43.2
100 "	55.3	59.8	51.8	49.8	41.6	36.0
1000 "	55.2	* *	54.8	45.3	39.8	31.1
10,000 "	63.0	53.0	51.4	43.7	39.2	23.0
100,000 "	56.6	56.5	50.2	42.8	34.0	17.0

**Not recorded









DATA AND RESULTS

Part 2: Delayed Quench Studies

Table 2 (a)

Specimens Austenitized at 2000°F; Delayed Quench 1200°F,
Time 15 seconds

Rockwell "C" Hardness Obtained by Tempering at
the Indicated Temperature and Time

Time at Tempering	600°F	800°F	1000°F	1200°F
10 sec.	50.1	48.5	44.0	46.9
100 "	48.5	43.3	39.3	31.8
1000 "	51.2	40.1	29.4	30.4
10,000 "	49.2	39.9	34.3	29.0

Table 2 (b)

Specimens Austenitized at 2000°F; Delayed Quench 1000°F,
Time 15 seconds

Rockwell "C" Hardness Obtained by Tempering at
the Indicated Temperature and Time

Time at Tempering	600°F	800°F	1000°F	1200°F
10 sec	42.6	40.4	37.3	34.1
100 "	45.1	42.1	34.8	30.1
1000 "	42.7	42.3	31.8	28.1
10,000 "	45.3	38.5	34.6	26.8

Table 2 (c)

Specimens Austenitized at 2000°F; Delayed Quench 800°F,
Time 15 seconds

Rockwell "C" Hardness Obtained by Tempering at
the Indicated Temperature and Time

Time at Tempering	600°F	800°F	1000°F	1200°F
10 sec	40.5	37.6	38.5	32.7
100 "	46.6	39.1	35.6	31.2
1000 "	44.3	37.5	34.8	29.7
10,000 "	44.3	39.3	31.7	25.6

Table 2 (d)

Specimens Austenitized at 2000°F; Delayed Quench 600°F,
Time 15 seconds

Rockwell "C" Hardness Obtained by Tempering at the Indicated Temperature and Time				
Time at Tempering	600°F	800°F	1000°F	1200°F
10 sec	45.9	44.8	43.4	43.8
100 "	45.6	43.3	42.0	38.0
1000 "	40.3	43.4	40.8	34.6
10,000 "	47.8	43.4	39.8	29.2

Suggestions for further Work

Further work can be done on the Tempering Behavior of this steel which can include:

- (1) Austenitizing at 1750°F and 1500°F and arresting the quench at the same temperatures as were used in this work for the austenitization at 2000°F, and then tempering at these same temperatures.
- (2) Studies of the normal tempering treatment for periods longer than is used in the present work -- about 1 week.
- (3) Studies of the microstructures obtained during each tempering cycle. This will serve to correlate the hardness values obtained with the structure at each cycle.
- (4) Supercooling the quenched structures to temperatures below the room temperature in liquid nitrogen before tempering. (However the steel seems to have high hardening ability and such refrigeration treatment may not have much effect.)

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